

A Survey of Contaminants on Four Delmarva Peninsula  
National Wildlife Refuges

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**Title:** A Survey of Potential Contaminants on Four Delmarva Peninsula

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**Abstract:** Personnel from the U.S. Fish and Wildlife Service, Annapolis Field Office (AFO), investigated the incidence of contaminants on Eastern Neck and Blackwater National Wildlife Refuges (NWRs), Maryland and Bombay Hook and Prime Hook NWRs, Delaware. Agriculture was the principal land use within and adjacent to these NWRs, and in addition to industry, provided a potential source of contaminants. Inorganics, organophosphate pesticides, organochlorines, and PCB levels were measured in sediments and toxicity was assessed with Microtox\ bacterial assays. Concentrations of inorganics were generally low when compared to contaminant studies from the Chesapeake Bay basin; and levels approximated those expected for a relatively pristine area, such as a wildlife refuge. In addition, organic compounds were detected in only 2 samples, both at the detection limit. Microtox\ assays indicated toxicity in 1 sediment sample from Blackwater, Prime Hook, and Eastern Neck NWRs; however, there was no apparent relationship between these toxicities, contaminant concentrations, and water quality parameters. However, lead and chromium levels at several sampling sites were elevated. Lead accumulations most likely resulted from the deposition of spent lead shot, although the source of chromium contamination was not known. Overall, samples from Eastern Neck NWR contained significantly lower levels of inorganics compared to the other refuges.

## ACKNOWLEDGEMENTS

Richard Bennett, former Environmental Contaminants Supervisor, designed this study and directed sample collections. Personnel from each of the refuges helped identify sampling locations and provided logistical support instrumental to successful field sampling. David Sutherland collected field samples and performed microtox\ assays. Peter Knight and Ilene Grossman helped collect field samples. John Moore, Patuxent Analytical Center, provided technical advice on catalog preparation. Monica Maghini wrote sections of this report detailing laboratory methods. John Morton and Monica Maghini assisted with data preparation and analysis. Robert Foley provided insightful comments on earlier drafts of this report, which greatly improved the document's quality.

## TABLE OF CONTENTS

	Page
Abstract . . . . .	i
Acknowledgements . . . . .	ii
Table of Contents . . . . .	.iii
List of Tables . . . . .	iv
List of Figures . . . . .	v
List of Appendices . . . . .	vi
Introduction . . . . .	1
The Refuges . . . . .	2
Materials and Methods . . . . .	10
Sample Collection . . . . .	10
Residue Analysis. . . . .	12
Analysis of Inorganic Data. . . . .	14
Organophosphate Pesticides and Organochlorines. . . . .	15
Microtox\ Bacterial Assay . . . . .	17
Results and Discussion. . . . .	18
Inorganics. . . . .	18
Organophosphate Pesticides and Organochlorines. . . . .	26
Microtox\ Bacterial Assay . . . . .	27
Summary and Conclusions . . . . .	27
Recommendations . . . . .	29
Literature Cited. . . . .	30
Appendices. . . . .	32



# LIST OF TABLES

	Page
Table 1. Percent recovery of inorganics in spiked sediment samples collected from Prime Hook (PH), Bombay Hook (BH), and Eastern Neck (EN) National Wildlife Refuges and a standard reference material. . . . .	13
Table 2. Duplicate analyses of inorganics (lg/g dry weight) in sediment samples collected from Prime Hook (PH) and Blackwater (BW) National Wildlife Refuges. . . . .	14
Table 3. Organophosphate pesticides analyzed in sediment samples collected from Eastern Neck, Blackwater, Bombay Hook, and Prime Hook National Wildlife Refuges. . . . .	16
Table 4. Organochlorines analyzed in sediment samples collected from Eastern Neck, Blackwater, Bombay Hook, and Prime Hook National Wildlife Refuges. . . . .	16
Table 5. Geometric means and ranges of inorganics (ppm dry weight) in sediments of 2 Chesapeake Bay National Wildlife Refuges (1990) with historical data from adjacent waterbodies (USEPA 1983). .	22

## LIST OF FIGURES

	Page
Figure 1. The locations of Eastern Neck (EN), Blackwater (BW), Bombay Hook (BH), and Prime Hook (PH) National Wildlife Refuges on the Delmarva Peninsula . . . . .	3
Figure 2. Locations of surface water and sediment sampling sites within Eastern Neck National Wildlife Refuge, Maryland, 1990-91 . . . . .	5
Figure 3. Location of surface water and sediment sampling sites within Blackwater National Wildlife Refuge, Maryland, 1990-91 . . . . .	6
Figure 4. Location of surface water and sediment sampling sites within Bombay Hook National Wildlife Refuge, Delaware, 1990-91 . . . . .	8
Figure 5. Locations of surface water and sediment sampling sites within Prime Hook National Wildlife Refuge, Delaware, 1990-91 . . . . .	11

## LIST OF APPENDICES

	Page
Appendix A. Water chemistry data collected with the Hydrolab water surveyor. . . . .	33
Appendix B. Inorganics concentrations and geometric means (ppm dry weight) for Eastern Neck, Blackwater, Bombay Hook, and Prime Hook National Wildlife Refuges . . . . .	36

## INTRODUCTION

The Delmarva Peninsula contains a mixture of urban centers, agricultural lands, woodlands, and both fresh and saltwater marshes. The network of coastal wetlands along the Delmarva Peninsula represents a rich biotic environment that is prized for its highly productive wildlife habitat. The principal cash crops on the Peninsula are corn, soybeans, wheat, and truck crops. These crops constitute a food source for migrating and wintering waterfowl. The Delmarva Peninsula is an important staging area for migratory birds and supports some of the highest concentrations of waterfowl in the Atlantic Flyway.

A series of National Wildlife Refuges (NWRs) was established on the Delmarva Peninsula to preserve habitat critical to migratory birds, and to enhance waterfowl populations. Both long-range and short-range sources of contaminants may affect NWRs on the Delmarva Peninsula. The industrial centers of Baltimore, Philadelphia, and Wilmington contribute pollutants to Chesapeake and Delaware Bay estuaries through long-range transport mechanisms. However, discharges from the few National Pollutant Discharge Elimination System (NPDES) permitted sites in communities surrounding the NWRs are not likely to contribute contaminants to the refuges (P. Janiga, Delaware Department of Natural Resources and Environmental Control (DENREC), and S. Luckman and E. Gertler, Maryland Department of the Environment, personal communications). Additional sources of contaminants, both on and adjacent to refuges, exist in runoff from agricultural fields treated with herbicides and pesticides. Agricultural practices are an

important management tool on NWRs. Farming maintains open fields and unharvested crops provide wildlife with a winter food source.

Because the U.S. Fish and Wildlife Service was responsible for identifying and evaluating potential pollutants and their sources on NWRs, the Annapolis Field Office (AFO) investigated potential contaminant levels at 4 Delmarva refuges. Sediments were selected as the environmental medium because: 1) sediments serve as a reservoir and source of contaminants to the water column and 2) sediments are an integral part of the aquatic environment, providing habitat for many aquatic organisms. Contaminants were surveyed at Eastern Neck, Blackwater, Bombay Hook, and Prime Hook NWRs during 1990-91. In addition, AFO personnel measured the toxicity of surface water and sediment with a microbial test and compared toxicity to contaminant loads.

#### THE REFUGES

Eastern Neck NWR is a 911 ha island at the mouth of the Chester River on the eastern shore of Chesapeake Bay (Fig. 1). Eastern Neck is in Kent County, Maryland, 8 km south of Rock Hall on Route 445. The refuge is a major feeding and resting area for waterfowl along the Chesapeake's eastern shore. The primary purpose of the refuge is to provide and enhance habitat for migrating and wintering waterfowl, and to provide optimum year-round habitat for the endangered Delmarva fox squirrel (Sciurus niger cinereus). Eastern Neck is comprised of cropland (25%), woodland (25%), and wetland (50%).

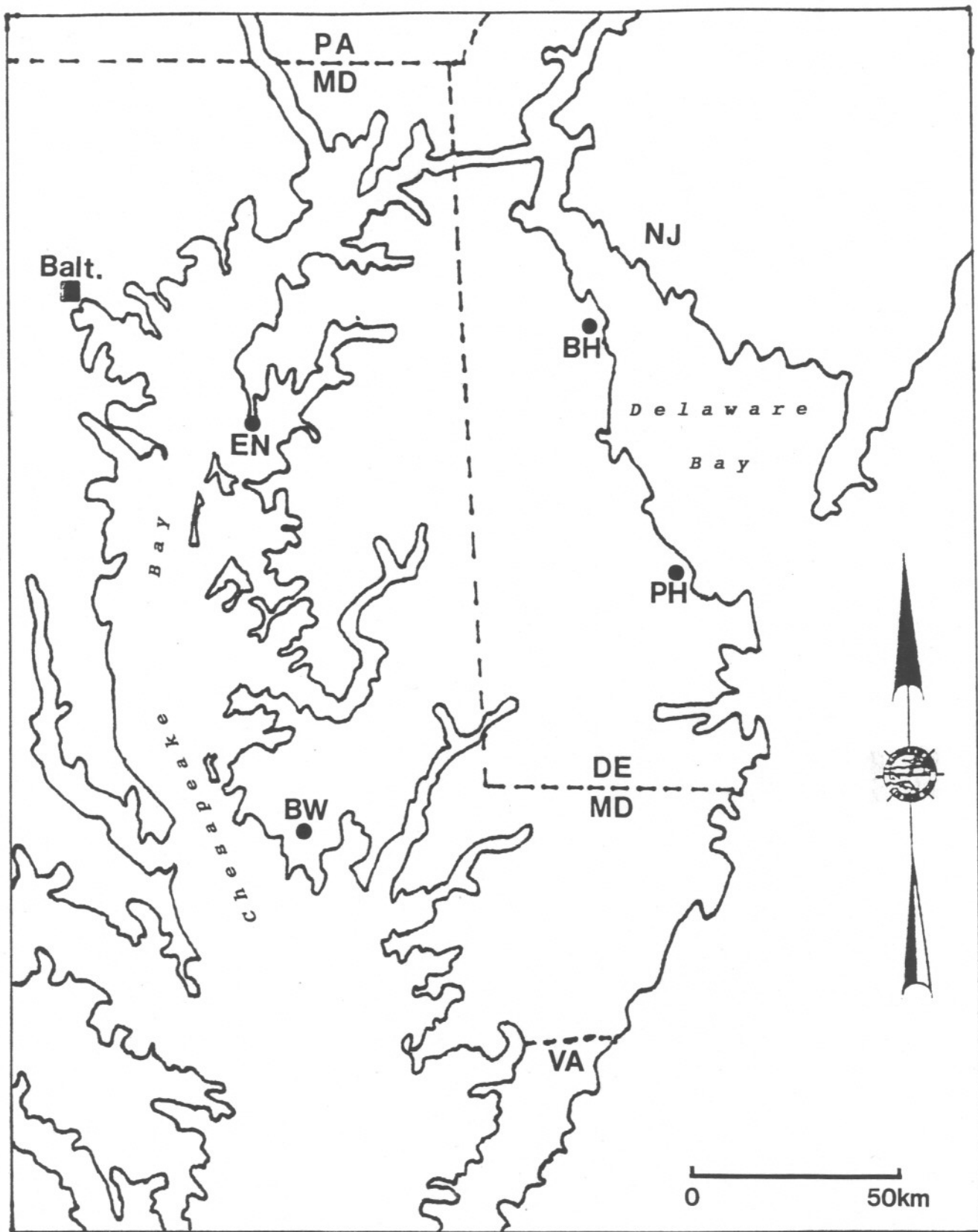


Figure 1. The locations of Eastern Neck (EN), Blackwater (BW), Bombay Hook (BH), and Prime Hook (PH) National Wildlife Refuges on the Delmarva Peninsula.

Sampling site 1 was in the flats at the northwest end of Eastern Neck Island (Fig. 2). Site 1 was west of Calfpasture Cove and 91 m north of the cove shoreline. This area received heavy waterfowl use in the fall. Site 2 was located in a channel in a drawdown wetland in the central portion of Eastern Neck NWR. Site 3 was in the northeast quadrant of the refuge, in a man-made pond east of Tubby Cove and a refuge access road. The remaining 3 sites were in embayments. The fourth sampling location was in Hail Creek. Site 5 was in Shipyard Creek, and site 6 was in Durdin Creek, an area of low waterfowl usage.

Blackwater NWR is on the eastern shore of Chesapeake Bay, 19.3 km south of Cambridge, in Dorchester County, Maryland (Fig. 1). Approximately 75% of the refuge's 6,929 ha are in estuarine habitats. The remainder of Blackwater's area is divided between palustrine forested wetlands, uplands, and croplands. The refuge's loblolly pine (*Pinus taeda*)-mixed hardwood forests provide habitat for the Delmarva fox squirrel. The fox squirrel population on Blackwater is one of the largest within the species' former range, with a stable population of approximately 550. Southern bald eagles (*Haliaeetus leucocephalus*) nest on the refuge and peregrine falcons (*Falco peregrinus*) inhabit the refuge during seasonal migrations.

Sampling site 1 was the western most, located in the Blackwater River at the Route 335 bridge (Fig. 3). Site 2 was upstream of a bridge over the Little Blackwater River, near a northern entrance to the refuge. Site 3 was in a tidal creek south of the refuge at Shorters Wharf. The fourth sampling location was in a drainage ditch at the mouth of Hudson Creek.

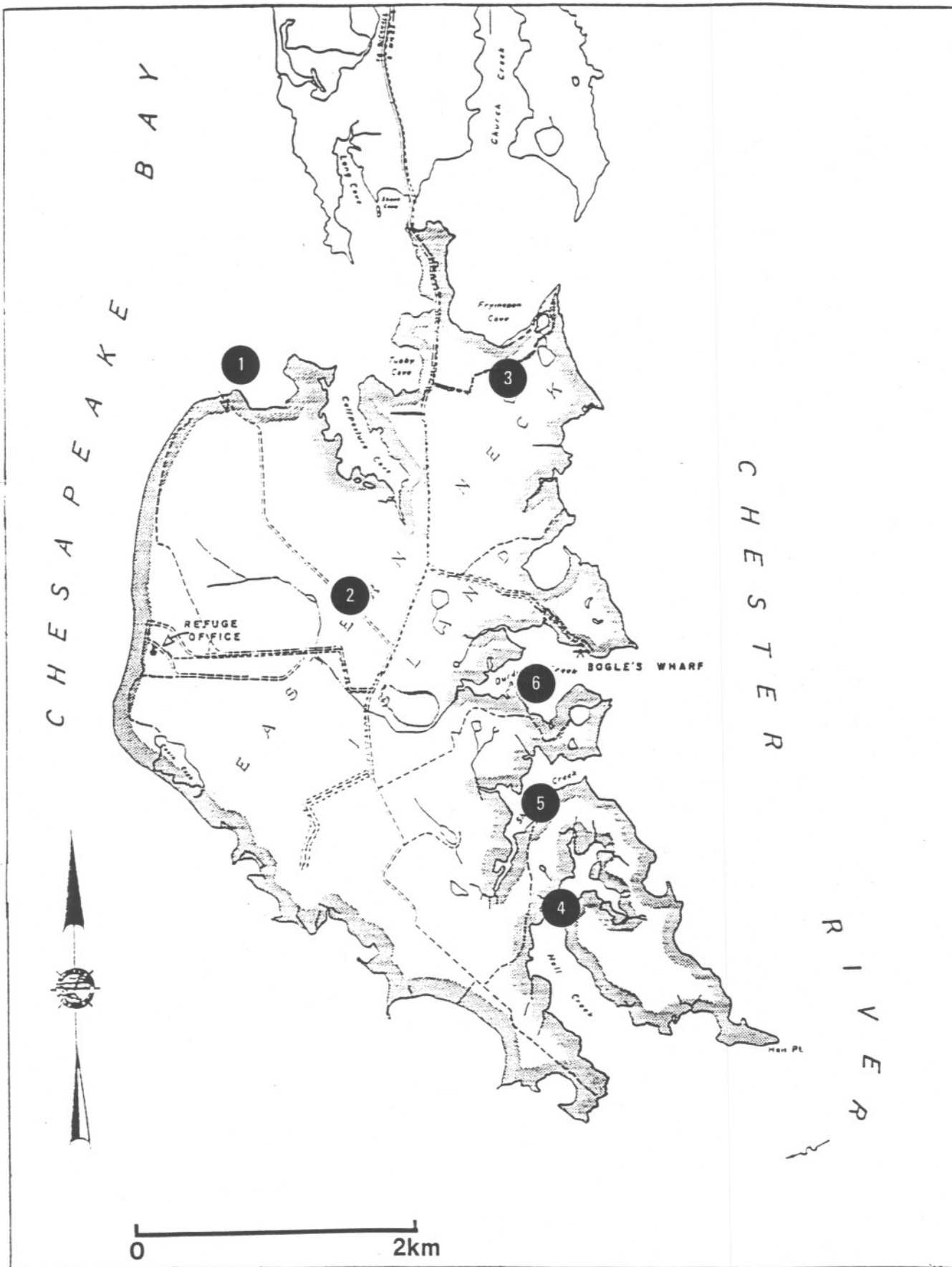


Figure 2. Location of surface water and sediment sampling sites within Eastern Neck National Wildlife Refuge, Maryland, 1990-91.



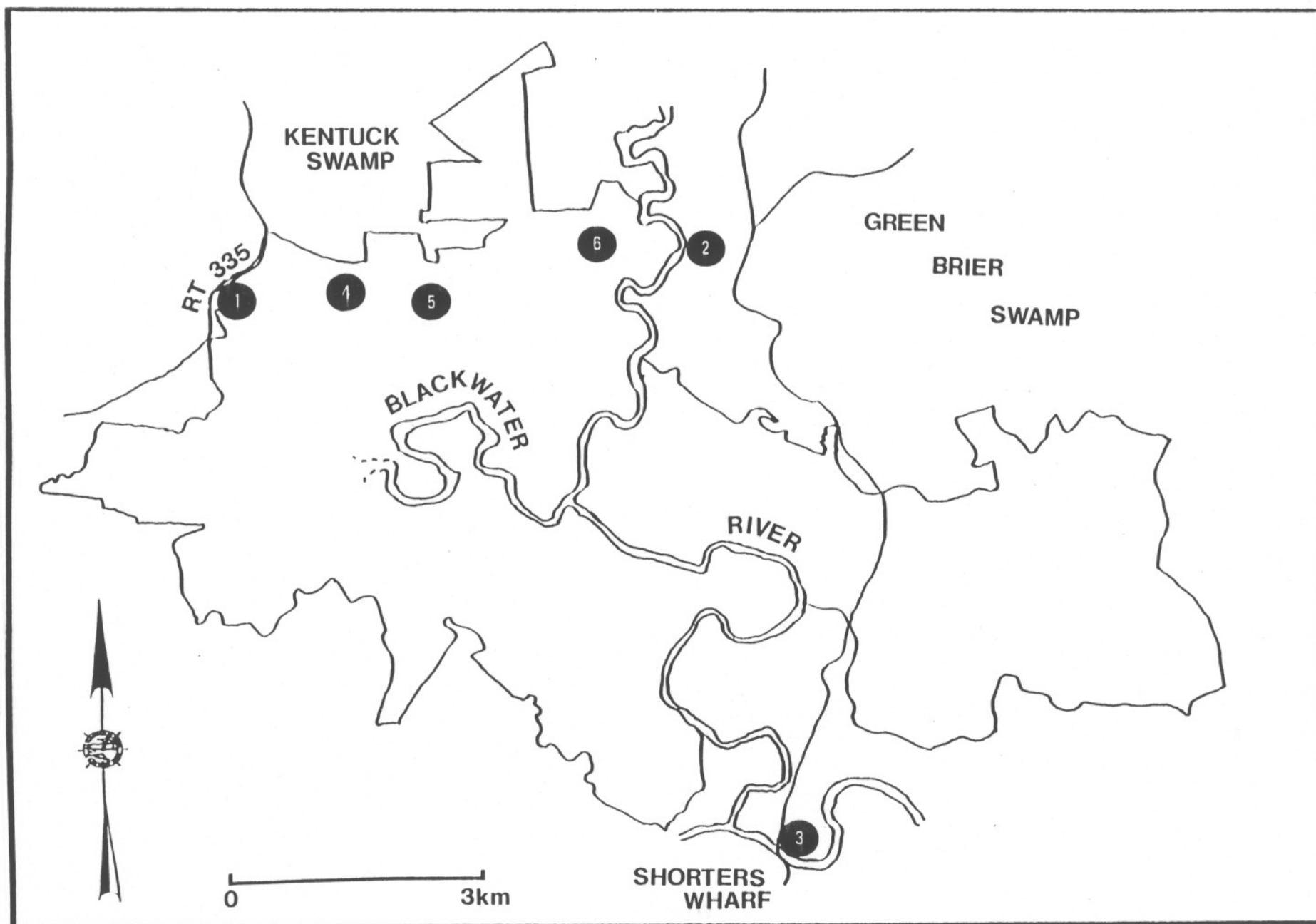


Figure 3. Location of surface water and sediment sampling sites within Blackwater National Wildlife Refuge, Maryland, 1990-91.

Sampling site 5 was in a ditch adjacent to pool 3, east of Gum Island.

Site 6 was in pool 2, west of the maintenance building and north of the dike road.

Bombay Hook NWR is located in Kent County, Delaware, 13 km southeast of Smyrna (Fig. 1). The refuge contains 6,120 ha of tidal marsh, mud flats, and tidal creeks and rivers. Bombay Hook also contains 399 ha of agricultural land, 445 ha of freshwater impoundments, and 299 ha of upland timber and brush. Dover Air Force Base previously owned a rifle range south of Herring Branch (Fig. 4). The land was added to Bombay Hook in the 1970's and refuge personnel recently learned of past contamination to the site. Apparently, Dover Air Force Base contributed potential contamination by disposing toxic materials and exploding ordnance at the range.

The primary goal of Bombay Hook is to provide and preserve habitat for migrating and nesting waterfowl, and for its endangered species (bald eagle and peregrine falcon). A secondary aim on Bombay Hook, as on other refuges is providing wildlife-oriented recreational opportunities, such as: environmental education, nature study, hunting, and fishing.

Sampling site 1 was in Hawkey Branch, east of the Route 9 bridge (Fig. 4). The second sampling location on Bombay Hook was in Finis Branch near the Route 9 bridge. Site 3 was downstream of site 2 on Finis Branch, and downstream of the bridge between Finis and Shearneck Pools. Site 4 was on the Leipsic River near Whitehall Landing. Sampling location 5 was at the confluence of Muddy Branch with Boat Gut. Sampling site 6 was on Herring

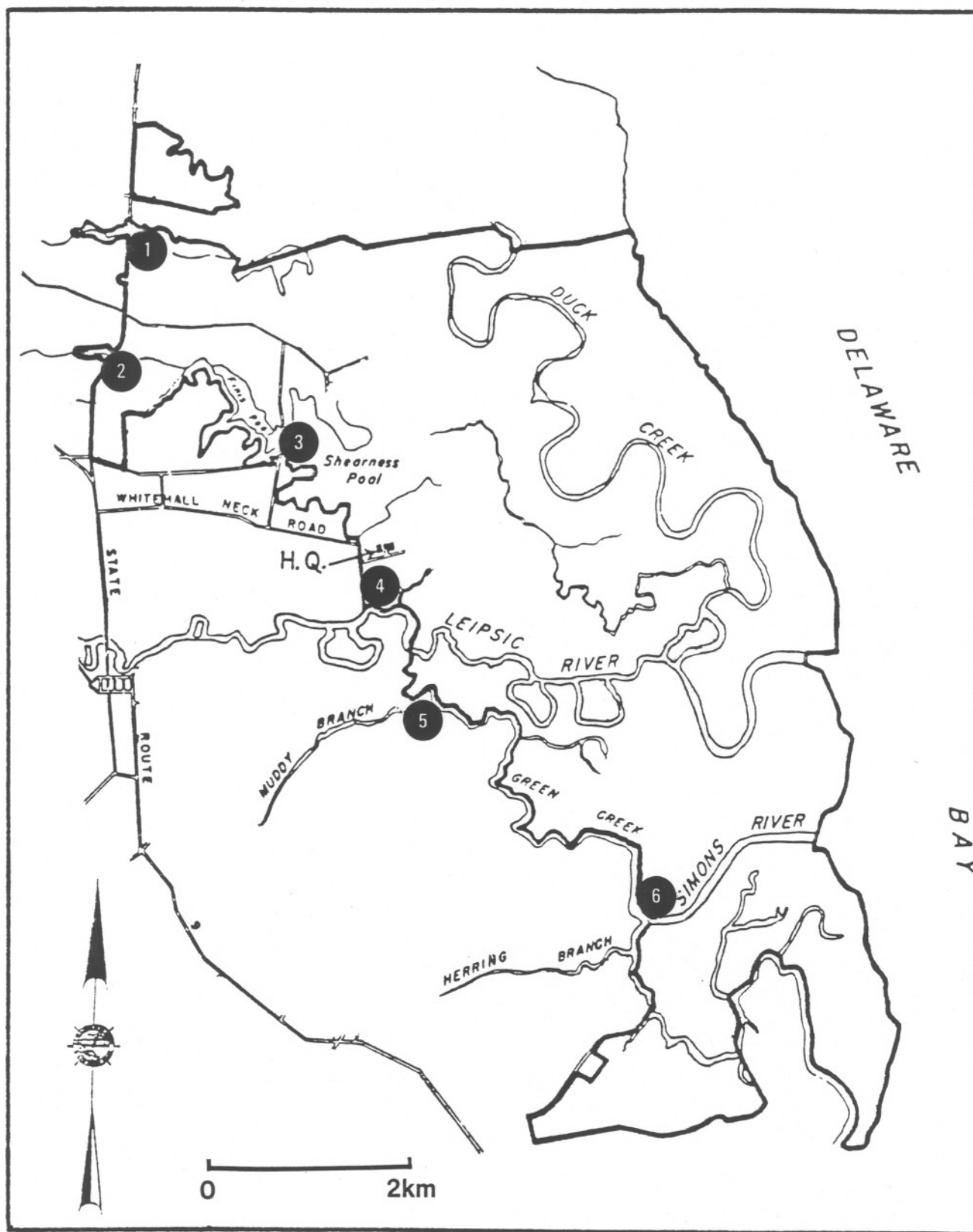


Figure 4. Location of surface water and sediment sampling sites within Bombay Hook National Wildlife Refuge, Delaware, 1990-91.

Branch, at the confluence of Green Creek and Simons River. Although nearest the former Dover Air Force Base property, site 6 was >1 km upstream of the contaminated area, and therefore did not measure potential contamination.

Prime Hook NWR is in Sussex County, Delaware, 8 km east of Milton (Fig. 1). The refuge is comprised of 3,568 ha acquired in fee title and 358 ha of flowage easements. Approximately 77% of Prime Hook is in wetland cover types. Other habitats include: 377 ha of agricultural lands, 262 ha of wooded uplands, 146 ha of grasslands, 89 ha of brush, and 8 ha of beach. Prime Hook initially was established to protect high value coastal wetlands from development. Recently, the objective of the refuge has broadened to include providing habitat for all indigenous flora and fauna, especially waterfowl and endangered species, such as the Delmarva fox squirrel.

Clifton vegetable cannery was a potential source of contamination to the refuge. Waste water from the plant's vegetable cleaning process sometimes entered a refuge tributary. The cannery processed sweet corn, peas, string beans, and lima beans during the May-October growing season. Clifton Canning Company was cited for 3 violations in 1987: 1) NPDES non-point discharge, 2) discharge of high temperature cooling water, and 3) an overflow of lagoon materials into Primehook Creek (USFWS memo). The cannery's spray irrigation system was improved after the 1987 violations, but DENREC ordered the correction of additional problems with the waste disposal system in August 1988 (Wilmington News-Journal 1988).

Sampling site 1 was in Prime Hook Creek downstream of Waples Pond (Fig. 5). This site received runoff from 2 sources; from Route 14 and a drainage pipe from Stix Tavern. Site 2 was farther downstream, where waste water runoff from the spray irrigation system at Clifton vegetable cannery sometimes emptied into Primehook Creek. Site 3 was downstream from the cannery tributary and an island, and upstream from Turner's boat launch. Site 4 was the northern most location. It was in Slaughter Creek at Primehook Neck Road. Sampling site 5 was in the southeast part of the refuge in the Broadkill River at Long Reach. Site 6 was in Broadkill Sound, 0.8 km south of Route 16.

## MATERIALS AND METHODS

### Sample Collection

Personnel from AFO collected sediment samples from 6 locations within each NWR between 24-27 July 1990 for chemical analyses. Additional samples of surface water and sediment were collected from the same locations between 3 March and 4 April 1991 for microbial assays. During both collection periods, a Hydrolab\ water quality surveyor recorded temperature, pH, dissolved oxygen, conductivity, and salinity of surface water at each sampling location (Appendix A). Technicians collected surface water samples with a teflon Kemmerer\ water sampling bottle. A 120 mL sample of surface water was collected for bacterial assays.

Technicians sampled sediments with a stainless steel petite ponar dredge. Three grab samples from each sampling site were mixed in a stainless steel

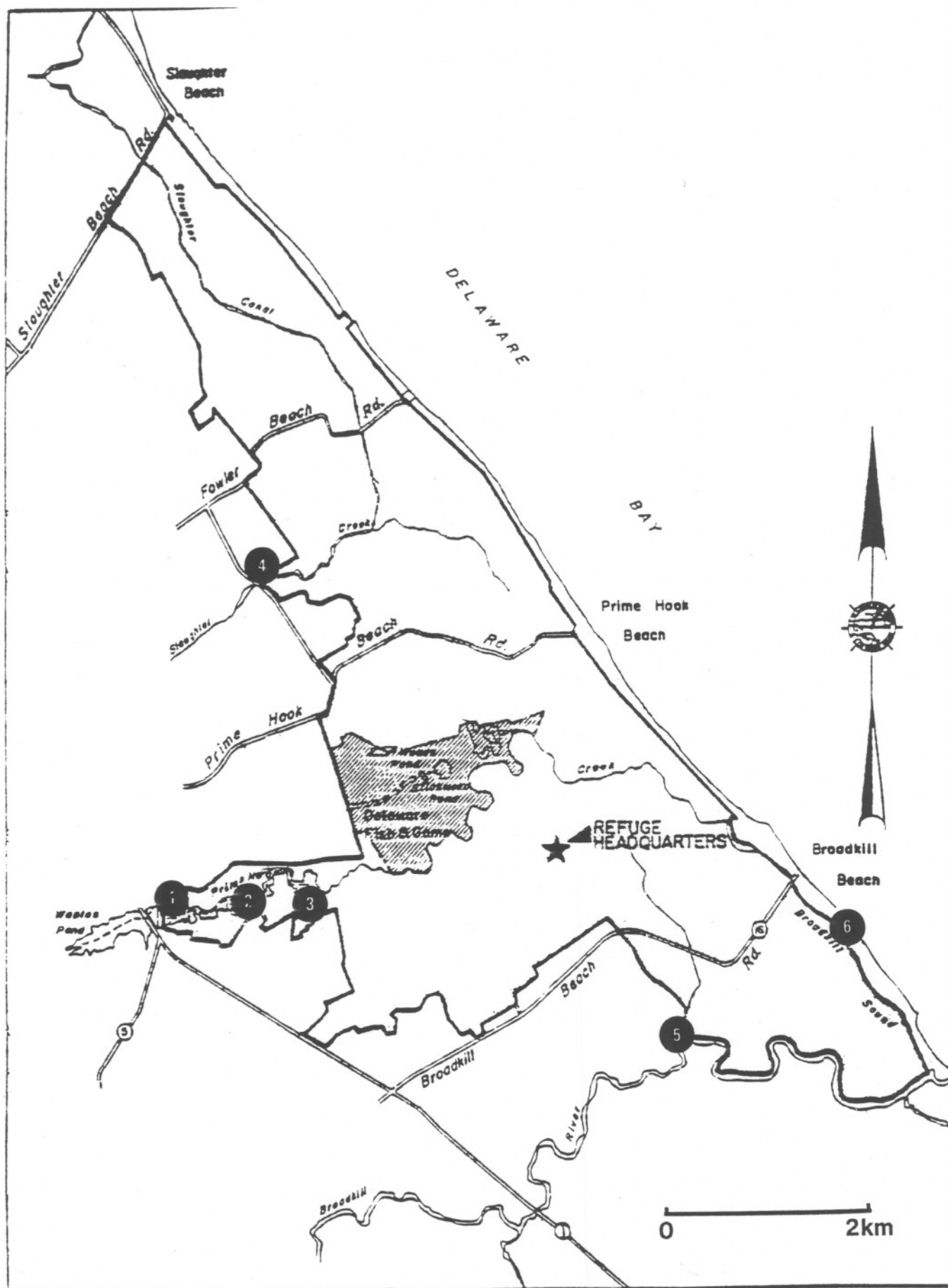


Figure 5. Location of surface water and sediment sampling sites within Prime Hook National Wildlife Refuge, Delaware, 1990-91.

mixing bowl and placed in chemically clean jars for inorganic, OP pesticide, and organochlorine analyses. Technicians placed additional composited sediment samples in 240 mL centrifuge bottles for bacterial assays of pore water. Both water and sediment samples were stored on ice in the field. Samples were frozen prior to inorganic, OP pesticide, and organochlorine analyses, while additional samples were refrigerated at 4°C prior to Microtox\ assay.

### Residue Analysis

Research Triangle Institute (RTI), North Carolina, analyzed the sediment samples for 20 inorganics and percent moisture content with Inductively Coupled Plasma Emission Spectroscopy (ICP). The samples were lyophilized to calculate per cent moisture. The samples were ground to approximately 100 mesh using a glass mortar and pestle. An aliquot of 0.25-0.5 g sediment was placed in a 120 mL teflon microwave vessel. One mL each HCl, HF, and HClO<sub>4</sub>, and 10 mL HNO<sub>3</sub> were added to the vessel. The vessel was capped and heated in a CEM microwave oven for 2 minutes at 120 watts, 3 minutes at 180 watts, and 10 minutes at 600 watts. The resulting residue was diluted to 100 mL with 5% HCl, and filtered through a Whatman 41 filter paper. Measurements of inorganics were made using a Leeman Labs Plasma Spec I sequential spectrometer with an HF resistant torch tip. The accuracy of the method, as determined by recovery of elements in spike, blank, and standard reference material was generally acceptable (Table 1). However, the recoveries of magnesium (Mg) and strontium (Sr) in spiked samples were as low as 19 and 22.8%, respectively. RTI did not report recoveries for aluminum (Al), barium (Ba), and iron (Fe) because

Table 1. Percent recovery of inorganics in spiked sediment samples collected from Prime Hook (PH), Bombay Hook (BH), and Eastern Neck (EN) National Wildlife Refuges and a standard reference material.

Element	% recovery-spiked samples			x % recovery-standard reference material (n=3)
	PH-2-S1	BH-2-S1	EN-2-S1	NIST 2704
Al	a	a	a	61
Sb	110	b	b	c
Ba	a	a	a	86
Be	94.5	101	99.5	c
B	103	113	101	c
Cd	105	108	103	98
Co	102	105	101	c
Cr	102	106	102	86
Cu	105	108	103	95
Fe	a	a	a	78
Pb	104	103	100	106
Mg	a	45	19	60
Mn	85	a	a	95
Mo	109	108	105	c
Ni	103	106	104	94
Ag	119	118	131	c
Sr	23.5	33.3	22.8	69
Sn	108	a	a	c
V	102	109	102	94
Zn	104	106	99	95

a % recovery not calculated because the spike to background ratio <1.

b no spiked sample analyzed.

c expected concentration unknown.

the spike to background ratio did not exceed 1. Four method blanks were analyzed. All of the results were below the limits of detection for the method. The precision of the method, as determined by duplicate analysis was acceptable (Table 2).



Table 2. Duplicate analyses of inorganics (lg/g dry weight) in sediment samples collected from Prime Hook (PH) and Blackwater (BW) National Wildlife Refuges.

Element	<u>PH-1-S1</u>		<u>PH-3-S1</u>		<u>BW-2-S1</u>	
	Sample	Dup.	Sample	Dup.	Sample	Dup.
Al	1460	1650	7740	4810	12900	14000
Sb	<50 <sup>a</sup>	<50	<50	<50	<50	<50
Ba	151	157	524	516	75.7	85.7
Be	<0.25	<0.25	2.35	2.50	1.46	1.50
B	<5.0	<5.0	13.1	15.3	28.9	19.9
Cd	<0.5	<0.5	0.833	1.11	<0.5	<0.5
Co	<15	<15	24.9	23.8	17.4	18.9
Cr	<3.0	<3.0	34.1	35.6	30.7	31.7
Cu	<3.0	<3.0	10.7	11.6	13.2	13.4
Fe	1520	1380	13100	13600	16500	17000
Pb	6.43	6.21	57.1	75.8	31.7	32.4
Mg	<100	<100	950	420	2590	2000
Mn	8.85	8.47	200	205	310	342
Mo	<4.0	<4.0	<4.0	<4.0	<4.0	<4.0
Ni	<4.0	4.50	25.3	25.9	21.1	22.2
Ag	<10	<10	<10	<10	<10	<10
Sr	7.43	8.55	49.3	35.4	44.5	54.0
Sn	<50	<50	<50	<50	<50	<50
V	<5.0	<5.0	41.3	41.2	50.5	52.0
Zn	<5.0	<5.0	200	196	71.2	80.3

<sup>a</sup> Concentrations with a "<" symbol represent samples recorded below the respective detection limit.

#### Analysis of inorganic data

Inorganic data were analyzed statistically to determine differences in individual inorganic concentrations between refuges and total inorganic levels between refuges. The absence of replicate samples from individual sample locations precluded the statistical comparison of inorganic concentrations within refuges. The variation in individual inorganic levels between refuges was tested with a series of ONEWAY ANOVAs (Analysis of Variance), and total inorganic loads between refuges were compared using the Kruskal-Wallis Test (SAS Institute 1988). Inorganics with

concentrations below detection levels at more than half the sampling locations (>12) were not analyzed statistically. To analyze data for the remaining inorganics, non-detects were assigned their detection levels to assure the level of contamination is not underestimated. Data were log10 transformed because of non-normal distributions and unequal variances within the data sets.

#### Organophosphate Pesticides and Organochlorines

Patuxent Analytical Control Facility (PACF) analyzed the samples for 25 OP pesticides (Table 3) using standard procedures (Patuxent Wildlife Research Center 1989). PACF used a 30 m megabore gas chromatography column packed with 7% cyanopropyl and 7% phenyl polysiloxane for the analysis, and a 10 g aliquot of sand was added to the sediment during extraction to prevent caking of the sediment. The results were recorded in lg/g wet weight, and the limit of detection for all the compounds was 0.5 lg/g wet weight. The accuracy of the method, as measured by spike and blank analyses, was acceptable. Spike recoveries were determined for 5 of the compounds: recoveries ranged from 105.6-127.8%. Recoveries of analytes from a procedural blank were below the 5.0 lg limit of detection for all of the analytes. Duplicate analyses were conducted on a sample from site 6 on Bombay Hook to measure the precision of the method. Analyte concentrations in both samples were recorded below the limits of detection.

Mississippi State Chemical Laboratory analyzed the sediment samples for 21 organochlorines (Table 4), total PCBs, and percent moisture content. Twenty grams of each sediment sample were extracted for organochlorine and

Table 3. Organophosphate pesticides analyzed in sediment samples collected from Eastern Neck, Blackwater, Bombay Hook, and Prime Hook National Wildlife Refuges.

Acephate	Azinphos-methyl	Chlorpyrifos-dursban
Coumaphos	Demeton	Diazinon
Dichlorovos	Dicrotophos	Dimethoate
Disulfoton	Dursban	EPN
Ethoprop	Famphur	Fensulfothion
Fenthion	Malathion	Methamidophos
Methyl parathion	Mevinphos	Monocrotophos
Parathion	Phorate	Turbufos
Trichlorfon		

Table 4. Organochlorines analyzed in sediment samples collected from Eastern Neck, Blackwater, Bombay Hook, and Prime Hook National Wildlife Refuges.

HCB	a-BHC	C-BHC
$\beta$ -BHC	d-BHC	Oxychlordan
Heptachlor Epoxide	C-Chlordane	t-Nonachlor
Toxaphene	o, p'-DDE	a-Chlordane
p, p'-DDE	Dieldrin	o, p'-DDD
Endrin	cis-nonachlor	o, p'-DDT
p, p'-DDD	p, p'-DDT	Mirex

PCB analyses. The aliquots soaked for 1 hour in acetone followed by 1 hour in hexane, with intermittent shaking. Combined extracts were centrifuged and decanted into a separatory funnel containing sufficient water to facilitate partitioning of residues into the hexane portion. The hexane portion was washed twice with water, concentrated, and transferred to a 1.6 g florisil mini-column. The aliquots were passed through the florisil column and eluted in 2 elution fractions. Fraction I, for analysis of 19 of the 21 compounds, consisted of 12 mL hexane followed by 12 mL of 1%

methanol in hexane. Fraction II, for analysis of dieldrin and endrin, consisted of 24 mL of 1% methanol in hexane. If additional cleanup was required to separate PCBs from other organochlorines in Fraction I, further chromatography on a Silicic acid column was performed. Residues in the Florisil fractions and the Silicic acid fractions were quantified by packed or megabore column, electron capture gas chromatography. The lower limit of detection for toxaphene and PCBs was 0.05 lg/g wet weight. The detection limit for the remaining compounds was 0.01 lg/g wet weight. Spike recoveries for 2 samples ranged from 80-110%. Spike analysis was not conducted for a-BHC, d-BHC, C-chlordane, toxaphene, total PCBs, and o,p'-DDD. None of the organochlorines were detected in the 3 reagent blanks and the matrix blank. Duplicate analysis was conducted for a duplicate sample from sampling site 1 on Eastern Neck. None of the compounds were detected above the detection limits in either sample.

#### Microtox\ bacterial assay

The Microtox\ microbial luminescence assay was performed on surface and sediment pore water using the manufacturer's procedures (Microbics Corporation 1991). Laboratory technicians centrifuged sediment samples at 3,500 rpm for 75 minutes and decanted the pore water in preparation for the bacterial assay. AFO staff measured the reduction in bioluminescence of *Photobacterium phosphoreum* with a Microtox\ Model 500 Toxicity Test System (Microbics Corp., Carlsbad, CA), a temperature-controlled biophotometer. Assays were completed within 24 hours of sample collection for Eastern Neck, Bombay Hook, and Prime Hook, while samples from Blackwater were analyzed 4 days after collection. The first assay was performed on samples

from Bombay Hook. Water samples were added to the *P. phosphoreum* and NaCl mixture to give final concentrations of 90, 45, 22.5, and 11.25%. Because toxicity levels appeared low in the samples from Bombay Hook, samples from the 3 remaining NWRs were analyzed at a 90% dilution (abbreviated procedure) to save time. Assay results were later compared with results of samples submitted for inorganic analysis. Distilled water and phenol were used as controls. Distilled water had  $EC_{50}$ s greater than 100%, while  $EC_{50}$ s of samples containing phenol ranged between 31.5-41.1% in a 5-minute assay.

## RESULTS AND DISCUSSION

### Inorganics

The interpretation of contaminant data for sediment samples poses several difficulties. First, the availability of contaminants in sediments to plants and animals depends upon the physical and chemical properties of the sediment and water column in the area. Variables such as particle size, pH, salinity, the redox potential, the presence of chelating agents, and the inorganic content of the substrate influence the mobility of contaminants (Olsen 1984). Particle size was not determined for the sediment samples, therefore, the results could not be normalized to eliminate the influence of large grained particles (i.e., sand). Secondly, only total concentrations of elements were reported; toxic valence states were not differentiated from nontoxic valence states (e.g.,  $Cr^{+6}$  and  $Cr^{+3}$ , respectively). In addition, although levels of contaminants in sediments are becoming more widely reported in the literature, the effects of these concentrations on biota (i.e., toxicity data) are not available for many

compounds. Unfortunately, toxicity data also contain inherent problems. A diminished confidence in toxicity tests results when the limited amount of data available necessitates comparing data from tests exposing organisms to differing environmental conditions. Interpreting intraspecies variation in test organism response leads to additional difficulties. Thirdly, the interaction of heavy metals in a system may either increase (additive or synergistic) or decrease (antagonistic) toxic effects to aquatic biota (Irwin 1989). Demayo et al. (1980) reported additive effects of lead (Pb) with mercury (Hg), copper (Cu), cadmium (Cd), and zinc (Zn) on aquatic life. Synergistic effects are more toxic than additive effects and have been documented for Pb and Cd (Demayo et al. 1980).

In the analyses of inorganics in sediments, concentrations are expressed in ppm dry weight (DW). Antimony (Sb) and tin (Sn) concentrations were not recorded above detection limits of 50 ppm and silver (Ag) was not detected above 10 ppm. Molybdenum (Mo) concentrations were recorded above the detection limit (4.0 ppm) in 2 out of 24 samples, and Cd concentrations were recorded above the detection limit (0.5 ppm) in 4 out of 24 samples. Therefore, Mo and Cd were not analyzed statistically. The results of inorganic analyses and the geometric mean value for inorganics at each NWR are presented in Appendix B.

Concentrations of individual inorganics did not differ significantly between refuges except Ba. Prime Hook (PH) sediment samples contained Ba in significantly greater concentrations (geometric mean = 313 ppm) than

Blackwater (129 ppm) and Eastern Neck (111 ppm) ( $P = 0.0117$ ). The highest Ba concentration (524 ppm) was detected at site PH-3.

Total inorganic loads on Eastern Neck were significantly lower than the other refuges ( $P = 0.001$ ), but loads did not differ significantly between the remaining refuges. The low concentration of inorganics detected on Eastern Neck probably results from a lack of upstream contaminant sources to the island, whereas NWRs bisected by waterways originating off refuge are at a greater contaminant risk. In comparison, each of the remaining NWRs was crossed by  $\geq 2$  waterways.

Although Prime Hook had the highest inorganic concentrations overall, sample location PH-1 had the lowest inorganic load of all Prime Hook locations, and the elimination of PH-1 in analyses would have greatly increased the average inorganic levels on Prime Hook. Except for Al, Ba, and Sr, PH-1 contained lower inorganic concentrations than all NWR sites; and of the 15 inorganics analyzed, 8 were below the detection limits.

Analyses indicated higher concentrations of inorganics at PH-2 and 3, compared to PH-1, an upstream site. The discrepancy between inorganic levels may be due to contaminants settling out in Waples Pond, upstream of PH-1, or from discharges from Clifton Cannery into the tributary assessed by PH-2. The cannery, adjacent to Prime Hook NWR, has violated its discharge permits in the past (Wilmington News-Journal 1988) and continued contamination is suspected (O'Shea, U.S. Fish and Wildlife Service, personal communication). Cannery related contaminants such as OP



pesticides and inorganics used in food processing were not analyzed adequately. For example, Sn is used in food packaging (Goyer 1986) and may be of concern because the 50 ppm detection limit in this study is higher than the maximum concentration (43 ppm) reported in a nation-wide sampling of sediments (U.S. Dept. Commerce 1988).

Additional trends are evident when comparing inorganic concentrations between sites on the remaining NWRs. On Eastern Neck (EN), levels of B, Cr, Cu, Pb, Ni, Sr, and Zn were highest at 2 embayments, sites EN-4 and 5. The 3 river sampling locations on Blackwater (BW) NWR (BW-1, 2, and 3) had higher levels of Al, B, Co, Mg, Mn, and Sr than at the remaining sample locations in drainage ditches (BW-4 and 5) and a pool (BW-6). On Bombay Hook (BH), site BH-3 contained the lowest concentrations of all inorganics except Ba. This site was in Finis Branch between Finis and Sheariness pools. The results of these within-refuge comparisons suggest higher inorganic levels in open-ended systems, such as creeks with a constant flow compared to closed systems, such as potholes and pools.

USEPA (1983) reported sediment data from Chesapeake Bay locations adjacent to NWRs in the current study (Table 5). The Chester River, a Chesapeake Bay tributary, flows along the eastern shore of Eastern Neck Island. Levels of Cd, chromium (Cr), Cu, Pb, and Zn were similar between those recorded on Eastern Neck and adjacent locations in the Chester River. This similarity provides greater confidence that the samples collected from Eastern Neck are representative of background inorganic levels in the area and that no point sources of inorganic contamination exist on the refuge.



Two reference sites were available for areas within 25 km of Blackwater NWR (Table 5). Tangier Sound is along the lower eastern shore, southeast of Blackwater. The sound is fed by the Nanticoke, Wicomico, Manokin, and Big Annemessex Rivers. The second available reference is the mouth of the Wicomico River. With the exception of Cr, levels at Blackwater were within ranges reported from adjacent reference sites. This suggests a potential point source of Cr contamination near Blackwater. Lead was another inorganic of potential concern on Blackwater. Although within the range reported for adjacent sites, the geometric mean for Pb at Blackwater exceeded the geometric means for the reference sites by  $\geq 56\%$ .

Table 5. Geometric means and ranges of inorganics (ppm dry weight) in sediments of 2 Chesapeake Bay National Wildlife Refuges (1990) with historical data from adjacent water bodies (USEPA 1983).

Element	Eastern Neck NWR <sup>a</sup> n = 6	Chester River <sup>b</sup> n = $\geq 10$	Blackwater NWR <sup>a</sup> n = 6	Tangier Sound <sup>b</sup> n = $\geq 10$	Wicomico River <sup>b</sup> n = $< 10$
Cd	<0.5 <sup>c</sup>	2 0.1-2	<0.5-0.9	1.1	1 0.1-5
Cr	22.8 10.7-43.6	19 2-110	36.0 27.5-49.5	27	9 2-20
Cu	7.6 <3.0-26.2	9 0.7-26	12.0 7.1-18.0	13	8 1-29
Pb	21.4 5.6-46.0	19 2-58	29.6 17.1-49.5	17	19 2-88
Zn	42.4 20.2-81.2	70 7-307	65.2 32.5-140	66	52 6-330

<sup>a</sup> Data reported in present study.

<sup>b</sup> Data from USEPA (1983).

<sup>c</sup> Concentrations with a "<" symbol represent samples recorded below the respective detection limit.

AFO completed 3 recent studies quantifying sediment toxicity to aquatic life and they serve as valuable references for the Chesapeake Bay region, linking contaminant levels to effects on biota. A contaminants study of Mason Neck NWR, Fairfax County, Virginia, investigated the possible causes of lesions in fish (Block 1990). Elevated levels of organic compounds were thought to be the major contributor to lesions, although concentrations of inorganics in sediment were also evaluated. Most of the inorganics analyzed appeared in higher concentrations in Mason Neck sediments than in the present study. Block (1990) noted that Pb concentrations, potentially contributed by a skeet range, were highest at the 2 sampling sites in Accotink Bay (82 and 69 ppm) and all fish collected there contained Pb. The highest Pb level recorded in the present study was 60 ppm at PH-4, and 2 other sites on Prime Hook contained Pb concentrations above 50 ppm.

The elevated Pb levels discussed for Prime Hook and Blackwater, and the high concentrations reported from the other NWRs support the findings of DiGiulio and Scanlon (1984), who described high levels of Pb in waterfowl from Chesapeake Bay. They attributed elevated Pb levels to the ingestion of lead shot and foods with elevated Pb. Because waterfowl hunting is a traditional activity on NWRs, the high levels of Pb probably result from an accumulation of spent lead shot in the sediment. With the introduction of steel shot, waterfowl are less likely to ingest lead pellets, however the persistence of Pb in the environment provides a reservoir for the transfer of Pb through the food chain.

In an AFO study to identify potential contaminant threats to Presquile NWR, Prince George County, Virginia, sediment samples from 1 of 4 James River sites proved toxic to aquatic organisms during bioassays (Nair 1990). Samples from the site, near Hopewell Wastewater Treatment Plant, were toxic to both Ceriodaphnia and P. phosphoreum. Nair (1990) indicated that toxicity at the treatment plant was due to the presence of total DDT, total PCBs, and elevated levels of arsenic (As) (6.85 ppm), Cu (167 ppm), Pb (70 ppm), and Zn (419 ppm). In comparison, organochlorines were not of concern in this study, and maximum Cu (26.2 ppm), Pb (60 ppm), and Zn (200 ppm) concentrations were less than those reported by Nair (1990) at the effluent site, but similar to those reported at the upstream control sites.

Goodbred and Block (1990) focused on inorganics in sediment and biota in Mattawoman Creek, Charles County, Maryland. The creek is a tributary to the tidal portion of the Potomac River. Outfalls from Indian Head Naval Ordnance Station were suspected sources of contamination to Mattawoman Creek. Sediment samples were collected both at the Marsh Island experimental site and an upstream control site and analyzed for inorganics. Significantly more lesions were reported by Goodbred and Block (1990) in fish at the Marsh Island experimental site compared to the control site. The authors hypothesized that elevated concentrations of As, Cu, and Pb (geometric means 4.6, 32.9, and 49.9 ppm, respectively) caused chronic health effects in fish. Arsenic levels were not reported for the Delmarva NWRs in the present study, but the range of geometric means for Cu (7.6-12.0 ppm) and Pb (21.4-31.2 ppm) were substantially lower than levels at

Marsh Island. However, the levels on the NWRs were greater than geometric mean Cu (6.4 ppm) and Pb (10.7 ppm) levels upstream of Marsh Island. Geometric mean Cr concentrations on the NWRs (33.6-36.0 ppm), except Eastern Neck (22.8 ppm), exceeded concentrations reported for Marsh Island (23.3 ppm) and were more than 4 times greater than levels at the control site upstream of Marsh Island (8.7 ppm). This supports the earlier concerns that Cr concentrations are elevated on the NWRs and may result from local contamination.

The Maryland Spoil Disposal Criteria Committee (1975) developed guidelines for the disposal of sediments containing selected inorganics. These criteria were established subjectively. The committee reviewed existing EPA data and compiled background values from various parts of Chesapeake Bay. The resulting criteria were set  $\geq 50\%$  above background levels. These criteria do not carry the force of law and therefore serve only as guidelines. Dredge material with inorganic concentrations exceeding these guidelines should be disposed of at a contained site rather than open water. Guidelines were prepared for Cd (2 ppm), Cr (80 ppm), Cu (80 ppm), Pb (100 ppm), and Zn (300 ppm). Concentrations of these inorganics in this study were never greater than two-thirds of the guideline values, except for Cr. The highest levels of Cr reported were 91 ppm at BH-5 and 75 ppm at PH-2.

The National Status and Trends Program (U.S. Dept. Commerce 1988) evaluated contaminant levels in estuaries throughout the United States. Chesapeake and Delaware Bays had intermediate levels of contaminants in comparison

with estuaries throughout the United States. After comparing levels of inorganics in Chesapeake and Delaware Bays, higher levels of As, Cr, Cu, and nickel (Ni) were reported in Chesapeake Bay with higher levels of Hg in Delaware Bay. Arsenic and Hg were not analyzed in this study, however, no significant differences were found in levels of Cr, Cu, and Ni between the Chesapeake and Delaware Bay refuges. These differences may be the result of inland sampling on the refuges, not normalizing the sediment data for the refuges, or site specific differences within the bays.

#### Organophosphate Pesticides and Organochlorines

All the samples and duplicates analyzed for OP pesticides were below the 0.5 ppm (WW) detection limit. The laboratory reported that the OP pesticide analyses were qualitative; that extraction parameters had not been optimized; and that recoveries had not been determined. Compounds were reported below the detection limit possibly because of poor method performance or because of major interferences with analyte in sample matrices.

Even though technicians collected sediment samples during the summer growing season, when the application of pesticides was greatest, the negative results of OP scans may be due to poor adsorption to sediments, and the short persistence of OP pesticides in the environment (N. Beyer, U.S. Fish and Wildlife Service, personal communication). An additional reason for negative results may be that Regional FWS policy prohibits the application of restricted pesticides on NWRs. Nevertheless, because sediments are not an appropriate media to evaluate organophosphates,

pesticide levels on the refuges originating from either on- or off-refuge sources have not been adequately assessed.

Organochlorine concentrations of sediment samples were below the 0.01 ppm detection limit with 2 exceptions. A degradation product of DDT, p, p'-DDE, was recorded at the 0.01 ppm detection limit for PH-4 and 6. Because of the few detections and low levels of organochlorines detected on the refuges, contamination from organochlorines is not considered a danger to trust resources.

#### Microtox\ bacterial assay

Microtox\ testing did not result in  $EC_{50}$ s <100% for any surface water sample. However, positive Microtox\ assay responses of sediment pore water samples, at sites: PH-1, 2, 3, and 4; EN- 2 and 3; and BW-4, 5, and 6 suggest toxic biological effects. After a detailed assay was performed on these samples and all Bombay Hook samples, the following had  $EC_{50}$ s <100% : BW-4 77.2%, EN-2 67.4%, and BH-3 33.2%. There was no apparent relationship between these toxicities, contaminant concentrations, and water quality parameters, indicating the presence of unknown contaminants or false toxicity readings in the analysis.

#### SUMMARY AND CONCLUSIONS

National Wildlife Refuges are established to preserve areas rich in wildlife habitat, and the existing nationwide network is important in maintaining current migratory wildlife and endangered species populations.

Because of the essential role of NWRs in the continued needs of a diversity of wildlife, it is expected that the refuges remain in a relatively pristine condition. This study confirmed that contaminant levels for most inorganics, OP pesticides, and organochlorines in the 4 NWRs investigated, were low. This was especially true of Eastern Neck, an island refuge. Of the more toxic heavy metals analyzed (Cd, Cr, Ni, Pb, and Zn), Cr and Pb exhibited elevated levels on Blackwater NWR when compared to data from the Chesapeake Bay watershed. In addition, although references from the Delaware Bay watershed were unavailable, Cr and Pb levels from some Prime Hook and Bombay Hook NWR sites exceeded levels on Blackwater NWR. Of the remaining inorganics, Mg and Sr were in some cases >2 times levels reported in other AFO studies and are possibly underestimated as suggested by the low recoveries in spiked samples. Antimony and Sn analyses had high detection limits (50 ppm) and toxic levels may not have been detected. Barium was the only inorganic that differed in concentrations between refuges, with higher levels detected on Prime Hook. Organophosphate pesticides were not detected and organochlorines were recorded twice at the detection limit (0.01 ppm WW). However, sediments may not have been an appropriate media to assess OP pesticide levels. Samples from 3 locations were acutely toxic to Microtox\ bacteria.



## RECOMMENDATIONS

Follow-up needs of this preliminary study are:

- To identify possible OP pesticide contamination of refuges from off-refuge areas. This will require cholinesterase analysis of fish and/or bird tissues.
- To assess the potential contaminant output of Clifton Cannery. Levels of certain inorganics were higher downstream of the cannery than at an upstream site, therefore additional sampling, including a direct measure of cannery outputs is warranted.
- To identify the valence states and sources of Cr contamination at Blackwater NWR, and determine whether Cr concentrations at Prime Hook NWR and Bombay Hook NWR are elevated. Because the toxicity of Cr is highly dependent on valence state, this information is necessary before the toxic effects of Cr levels documented in this study can be assessed.
- To determine the extent of Pb contamination in sediments of NWRs. Because waterfowl hunting is an historic use of NWRs, spent shot often accumulates in sediments. The fate of this shot, mobilization of Pb from refuge sediments, and strategies to reduce the threat of this problem need to be defined.
- To quantify the extent of potential contamination within the portion of Bombay Hook formerly owned by Dover Air Force Base. The potential for contamination at this site is high, and this study did not assess contaminants contributed by the site.



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## APPENDICIES

## APPENDIX A

Table A-1. Physical/Chemical data recorded at the time of ICP sample collection. Units are as follows: temperature - #C, dissolved oxygen - mg/L, conductivity - millimhos/cm, and salinity - ppt.

Eastern Neck NWR  
7/24/90

Site	EN-1	EN-2	EN-3	EN-4	EN-5	EN-6
Time	NR <sup>a</sup>	NR	NR	NR	NR	NR
Water Temp.	26.16	25.66	28.43	26.58	27.00	27.67
pH	8.62	5.53	6.53	7.39	7.52	7.50
D.O.	7.10	1.52	1.10	6.41	6.00	6.90
Conductivity	10.79	0.07	0.02	13.31	13.25	13.25
Salinity	6.70	0.00	0.00	7.40	7.30	7.30

Blackwater NWR  
7/27/90

Site	BW-1	BW-2	BW-3	BW-4	BW-5	BW-6
Time	NR	14:20	14:50	NR	11:00	NR
Water Temp.	29.37	29.50	28.50	27.65	26.00	24.57
pH	7.31	9.15	7.10	6.44	5.20	6.16
D.O.	7.30	10.60	5.82	7.64	5.23	4.58
Conductivity	2.62	3.61	8.79	0.61	0.64	0.17
Salinity	0.90	1.50	4.60	0.00	0.00	0.00

Bombay Hook NWR  
7/25/90

Site	BH-1	BH-2	BH-3	BH-4	BH-5	BH-6
Time	NR	NR	NR	NR	NR	NR
Water Temp.	24.60	22.19	24.43	27.66	27.56	27.66
pH	6.68	6.71	6.36	7.39	6.85	7.39
D.O.	5.01	0.74	1.21	5.24	2.95	5.24
Conductivity	14.14	0.26	0.15	25.80	21.80	25.80
Salinity	7.90	0.00	0.00	15.60	12.90	15.60

Prime Hook NWR  
7/26/90

Site	PH-1	PH-2	PH-3	PH-4	PH-5	PH-6
Time	NR	NR	NR	NR	NR	NR
Water Temp.	27.23	26.92	27.62	24.74	27.14	27.24
pH	6.92	6.58	6.79	6.87	6.79	6.88
D.O.	6.89	4.01	5.90	9.30	5.90	4.06
Conductivity	0.12	0.13	0.11	0.14	0.11	17.30
Salinity	0.00	0.00	0.00	0.00	0.00	10.00

<sup>a</sup> Not recorded.

Table A-2. Physical/Chemical data recorded at the time of Microtox\ sample collection. Units are as follows: temperature - #C, dissolved oxygen - mg/L, conductivity - millimhos/cm, and salinity - ppt.

Eastern Neck NWR  
4/1/91

Site	EN-1	EN-2	EN-3	EN-4	EN-5	EN-6
Time	08:55	11:30	11:00	09:20	09:45	10:00
Water Temp.	9.85	13.60	12.33	9.99	9.44	9.97
pH	10.66	8.65	10.81	10.68	10.85	10.83
D.O.	10.21	7.44	9.42	9.55	10.56	9.93
Conductivity	12.33	0.10	0.32	12.47	12.46	11.73
Salinity	6.80	0.00	0.00	6.80	6.80	6.40

Blackwater NWR  
4/4/91

Site	BW-1	BW-2	BW-3	BW-4	BW-5	BW-6
Time	08:25	09:15	12:30	11:00	10:30	10:05
Water Temp.	12.08	11.92	13.70	12.42	11.16	12.61
pH	9.71	9.75	9.34	9.51	8.45	9.14
D.O.	8.65	8.35	8.85	8.29	4.32	7.06
Conductivity	1.21	0.31	4.80	0.31	0.07	0.12
Salinity	0.10	0.00	2.20	0.00	0.00	0.00

Bombay Hook NWR  
3/11/91

Site	BH-1	BH-2	BH-3	BH-4	BH-5	BH-6
Time	09:00	10:00	09:30	13:05	12:45	12:15
Water Temp.	6.65	4.76	4.50	6.62	5.97	5.96
pH	11.16	11.85	11.79	11.33	11.27	11.15
D.O.	4.78	5.11	9.81	9.89	9.61	10.75
Conductivity	0.95	0.21	0.19	9.97	11.15	14.66
Salinity	0.00	0.00	0.00	5.30	6.00	8.20

Prime Hook NWR  
3/18/91

Site	PH-1	PH-2	PH-3	PH-4	PH-5	PH-6
Time	10:25	11:15	11:00	10:00	11:45	12:45
Water Temp.	9.02	9.11	9.13	9.27	6.69	7.08
pH	9.17	9.45	8.50	9.80	10.87	9.55
D.O.	11.99	10.81	11.96	9.58	10.80	10.93
Conductivity	0.12	0.14	0.13	0.16	34.30	31.30
Salinity	0.00	0.00	0.00	0.00	21.30	19.30

## APPENDIX B

Table B-1. Concentrations of inorganics and geometric means (ppm dry weight) for Eastern Neck (EN) National Wildlife Refuge sediment samples\*.

ELEMENT	EN-1	EN-2	EN-3	EN-4	EN-5	EN-6	GEOMEAN
Al	2550	8330	5280	15000	9850	4280	6434
Sb	<50.0*	<50.0	<50.0	<50.0	<50.0	<50.0	-
Ba	51.8	232	116	124	194	55.0	110.7
Be	0.35	1.02	0.67	1.10	1.26	0.62	0.8
B	8.7	10.6	7.9	33.6	33.6	15.8	15.3
Cd	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	-
Co	<15.0	<15.0	<15.0	16.4	<15.0	<15.0	15.2
Cr	10.7	28.4	18.0	34.0	43.6	17.1	22.8
Cu	4.62	<3.0	<3.0	26.2	19.2	9.46	7.6
Fe	3650	6070	7760	15200	15500	6040	7908
Pb	5.63	20.4	21.4	46.0	35.6	23.5	21.4
Mg	2150	620	650	2120	1420	615	1082
Mn	59.9	214	133	234	210	69.8	134.2
Mo	<4.0	<4.0	<4.0	<4.0	<4.0	<4.0	-
Ni	5.31	9.97	6.0	30.8	24.4	11.3	11.8
Ag	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	-
Sr	5.33	15.7	12.8	25.9	21.6	6.88	12.7
Sn	<50.0	<50.0	<50.0	<50.0	<50.0	<50.0	-
V	17.5	45.2	33.4	8.2	67.6	28.1	27.3
Zn	20.2	37.0	34.6	81.2	79.0	34.9	42.4
% moisture	29.2	50.7	53.9	74.9	58.7	42.5	

\* Measured concentration was less than detection limit.



Table B-2. Concentrations of inorganics and geometric means (ppm dry weight) for Blackwater (BW) National Wildlife Refuge sediment samples\*.

ELEMENT	BW-1	BW-2	BW-3	BW-4	BW-5	BW-6	GEOMEAN
Al	16600	12900	11700	3350	5420	6470	8155
Sb	<50.0*	<50.0	<50.0	<50.0	<50.0	<50.0	-
Ba	131	75.7	122	96.5	206	196	129.5
Be	2.28	1.46	1.55	2.38	0.792	1.40	1.6
B	29.8	28.9	36.5	10.6	15.7	25.9	22.7
Cd	0.90	<0.50	<0.50	<0.50	<0.50	<0.50	-
Co	29.4	17.4	16.2	<15.0	<15.0	<15.0	17.4
Cr	27.5	30.7	43.5	35.1	34.2	49.5	36.0
Cu	12.2	13.2	14.0	7.12	10.6	18.0	12.0
Fe	20100	16500	18600	21400	7800	13500	15506
Pb	49.5	31.7	25.4	17.1	20.3	48.8	29.6
Mg	4130	2590	2460	160	370	460	946
Mn	232	310	520	97.8	77.3	192	194.6
Mo	<4.0	<4.0	<4.0	<4.0	<4.0	<4.0	-
Ni	30.1	21.1	22.7	14.2	12.4	24.5	19.9
Ag	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	-
Sr	87.5	44.5	32.9	5.72	9.22	12.1	20.8
Sn	<50.0	<50.0	<50.0	<50.0	<50.0	<50.0	-
V	42.4	50.5	70.8	93.2	63.7	83.7	65.0
Zn	140	71.2	61.4	41.6	32.5	92.8	65.2
% moisture	87.1	89.5	82.4	44.4	48.0	79.9	

\* Measured concentration was less than detection limit.

Table B-3. Concentrations of inorganics and geometric means (ppm dry weight) for Bombay Hook (BH) National Wildlife Refuge sediment samples\*.

ELEMENT	BH-1	BH-2	BH-3	BH-4	BH-5	BH-6	GEOMEAN
Al	11800	15400	1370	17700	17600	3620	8092
Sb	<50.0*	<50.0	<50.0	<50.0	<50.0	<50.0	-
Ba	212	396	169	160	259	65.2	184.0
Be	1.32	2.02	0.266	1.53	1.7	0.38	0.9
B	23.6	17.5	<5.0	52.5	38.2	21.4	21.1
Cd	<0.50	0.64	<0.50	<0.50	<0.50	<0.50	-
Co	15.0	22.5	<15.0	22.8	20.8	<15.0	18.2
Cr	41.0	63.9	5.31	62.8	91.2	18.0	33.6
Cu	14.0	18.6	<3.0	13.0	26.0	3.98	10.1
Fe	29500	30400	4070	30800	37900	7460	17798
Pb	36.9	46.0	7.54	32.0	53.4	8.85	24.1
Mg	2050	1030	<100	4010	4280	828	1201
Mn	204	579	41.4	461	539	85.0	216.6
Mo	<4.0	<4.0	<4.0	<4.0	<4.0	<4.0	-
Ni	19.0	31.4	<4.0	30.2	34.3	8.0	16.4
Ag	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	-
Sr	29.0	30.6	11.9	46.7	53.5	12.6	26.3
Sn	<50.0	<50.0	<50.0	<50.0	<50.0	<50.0	-
V	68.7	100	10.1	86.2	102	24.2	49.5
Zn	194	123	9.21	102	159	26.2	67.4
% moisture	78.3	67.0	31.2	78.0	69.0	63.1	

\* Measured concentration was less than detection limit.

Table B-4. Concentrations of inorganics and geometric means (ppm dry weight for Prime Hook (PH) National Wildlife Refuge sediment samples\*.

ELEMENT	PH-1	PH-2	PH-3	PH-4	PH-5	PH-6	GEOMEAN
Al	1460	13100	7740	11200	13700	15800	8430
Sb	<50.0*	<50.0	<50.0	<50.0	<50.0	<50.0	-
Ba	151	367	524	349	287	323	313.0
Be	<0.25	1.91	2.35	2.49	1.52	1.44	1.4
B	<5.0	26.1	13.1	28.3	53.6	47.5	22.3
Cd	<0.50	<0.50	0.88	0.61	<0.50	<0.50	-
Co	<15.0	22.4	24.9	20.3	15.9	17.7	19.0
Cr	<3.0	74.8	34.1	62.5	66.5	64.5	35.7
Cu	<3.0	9.29	10.7	21.2	9.07	18.9	10.1
Fe	1520	22400	13100	24600	27100	24300	13904
Pb	6.43	31.1	57.1	60.0	25.6	52.5	31.2
Mg	<100	2450	950	1830	3180	3510	1297
Mn	8.85	253	200	257	276	290	144.8
Mo	<4.0	<4.0	<4.0	<4.0	4.09	<4.0	-
Ni	<4.0	29.7	25.3	30.4	27.3	26.7	20.1
Ag	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0	-
Sr	7.43	42.6	49.3	38.1	59.7	64.1	36.3
Sn	<50.0	<50.0	<50.0	<50.0	<50.0	<50.0	-
V	3.9	94.1	41.3	75.3	84.6	79.6	44.4
Zn	<5.0	95.1	200	179	63.1	132	72.2
% moisture	21.0	79.4	88.6	74.6	39.0	64.0	

\* Measured concentration was less than detection limit.



